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Indian Standard

POLYVINYL ALCOHOL FOR SIZING OF PAPER — SPECIFICATION

भारतीय मानक

कागज-चिक्कण के लिए पॉलिविनायल एल्कोहल — विशिष्टि

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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TO
IS 12715 : 1989 POLYVINYL ALCOHOL FOR SIZING OF PAPER — SPECIFICATION

[*Page* 1, *Table* 1, *Sl No.* (vi), *col* 3] – Substitute '1.0' *for* '0.5'.

(CHD 15)

Reprography Unit, BIS, New Delhi, India

Paper and Its Products (Excluding Packaging Materials) Sectional Committee, CDC 15

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 1 May 1989, after the draft finalized by the Paper and Its Products (Excluding Packaging Materials) Sectional Committee had been approved by the Chemical Division Council.

Polyvinyl alcohol is obtained as a product of hydrolysis of polyvinyl acetate. Conventionally, currency paper and high quality speciality papers have been surface sized with gelatin for many years. Though gelatin contributes significantly towards durability and printability, it can easily be degraded in the extraction process resulting in considerable variation in quality, ultimately affecting the quality of the currency paper or speciality paper. Polyvinyl alcohol on the other hand forms a continuous, non-crystalline, strong and elastic film which has a high tensile strength. The paper surface sized with polyvinyl alcohol also has higher double fold strength and low porosity and has resistance to acid and alkali. These properties contribute to durability and smoothness of the paper and its improved printability.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYVINYL ALCOHOL FOR SIZING OF PAPER — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements **and** method of sampling and test for polyvinyl alcohol for sizing of paper.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 1060 (Part 3) : 1969	Methods of sampling and test for paper and allied products : Part 3
IS 4661: 1986	Glossary of terms used in paper trade and industry (*first revision*)
IS 4905 : 1968	Methods for random sampling
IS 11087 : 1986	Paper for magnetic ink character recognition cheque printing (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 4661 : 1986 shall apply.

4 REQUIREMENTS

4.1 Description

The material shall be in the from of granules/ powder of pale yellow colour and free from offensive odour.

4.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Polyvinyl Alcohol for Sizing of Paper

(*Clauses* 4.2, 7.1 *and* 5.1.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Moisture content, percent by mass, *Max*	6.0	A
ii)	*p*H	6.5-7.5	B
iii)	Viscosity at 20°C, cP, *Min*	28	C
iv)	Bulk density, g/l	0.53-0.62	D
v)	Insoluble matter, percent by mass	Nil	B
vi)	Total ash (Na_3O), percent by mass, *Max*	0.5	F
vii)	Degree of hydrolysis, mole percent, *Min*	99	G

4.3 Performance Test

4.3.1 Papers sized with polyvinyl alcohol shall not have an offensive odour.

4.3.2 *Wax Pick Test*

Paper conforming to IS 11087 : 1986 coated with the material, when tested in accordance with **8** of IS 1060 (Part 3) : 1969 shall show no pick on 18 A wax.

5 PACKING, MARKING AND STORAGE

5.1 Packing

The material shall be packed in well-defined wide mouth container with tight fitting lids.

5.2 Marking

Each container shall be marked legibly with the following information:

a) Name of the material, with specific mention that the material is for paper sizing;
b) Name and address of the manufacturer;
c) Minimum net content;
d) Batch or code number; and
e) Date of manufacture/packing.

5.3 Storage

The material shall be stored in a cool and dry place.

6 SAMPLING

6.1 The method of drawing representative samples of the material and the criteria for determining conformity of the material to the requirements of this specification shall be as prescribed in Annex H.

7 TEST METHODS

7.1 Tests shall be conducted by the methods prescribed in Annex A to G and IS 1060 (Part 3) : 1969. References to relevant methods is given in col 4 of Table 1 and **4.3.2.**

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which effect the results of analysis.

ANNEX A

[*Table* 1, *item* (i) and *Clause* 7.1]

DETERMINATION OF MOISTURE CONTENT

A-1 APPARATUS

A-1.1 Oven, capable to maintain a temperature of 140 to 150°C.

A-1.2 Balance, sensitivity of 0.1 mg or better.

A-2 PROCEDURE

A-2.1 Weigh accurately 10 to 12 g of the sample in a covered glass weighing dish. Remove the cover and heat the bottle and its contents in an oven at 140 to 150°C for at least 4 hours. Replace the cover, cool in a desiccator and weigh to constant mass. Repeat drying, cooling and weighing until constant mass is obtained.

A-3 CALCULATION

$$\text{Moisture, percent by mass} = \frac{100\,(M_1 - M_2)}{M_1}$$

where

M_1 = mass in g of the sample before heating, and

M_2 = mass in g of the sample after heating.

ANNEX B

[*Table* 1, *item* (ii) and *Clause* 7.1]

DETERMINATION OF *p*H

B-1 APPARATUS

B-1.1 *p*H Meter, with suitable electrode.

B-2 PROCEDURE

B-2.1 Dissolve 1 g of the powdered sample by shaking after careful addition to a small quantity of warm, recently boiled, water in a stoppered flask of chemically resistant glass, and dilute the volume to 100 ml with recently boiled and cooled water. After shaking and allowing to cool to room temperature, determine the *p*H value of the solution by using the *p*H meter. Care shall be taken throughout the operation to avoid absorption of carbon dioxide from the air.

ANNEX C

[*Table* 1, *item* (iii) and *Clause* 7.1]

DETERMINATION OF VISCOSITY

C-0 GENERAL

C-0.1 The viscosity of a sample of the polyvinyl alcohol is determined at 20°C with a U-tube viscometer.

C-1 APPARATUS

C-1.1 Viscometer

Any type of glass capillary viscometer should normally be suitable. It is essential that the time of efflux should be within the accurate range of the instrument chosen. The viscometer should be calibrated in metric units, that is, centistokes.

C-1.2 Thermostatic bath maintained at 40.0 ± 0.2°C.

C-2 PROCEDURE

C-2.1 Place 1.5 g of the powdered sample in a corked bottle or flask, and add 105 ml of cold water. Let it swell and dissolve, then pour the liquid through a funnel, loosely plugged with cotton wool into the viscometer and allow to stand in the thermostatic bath for 15 minutes before taking the measurement as described in **C-2.2.**

C-2.2 Apply suction or pressure to bring the liquid level to a point about 5 mm above the upper timing mark. Release the suction or pressure and record the flow time taken for the bottom meniscus to pass from the top edge of the upper timing mark and the top edge of the lower timing mark. Record the thermometer readings at the beginning of, during and at the end of the timed flow. Repeat the measurement of flow time immediately without emptying or recharging until two successive flow times agree within 0.2 percent.

C-3 CALCULATION

C-3.1 Express the results either as the kinematic viscosity in centistokes or as the dynamic viscosity in centipoises at 12.5 percent concentration and 20°C:

$$\text{Kinematic viscosity, } v = C\,t$$

where

C = factor for the viscometer given in its calibration certificate, and

t = mean flow time, in seconds.

$$\text{Dynamic viscosity, } n = v\sigma$$

where

v = kinematic viscosity; and

σ = density measured at the same temperature, in g/cm^3.

ANNEX D

[*Table* 1, *item* (iv) and *Clause* 7.1]

DETERMINATION OF BULK DENSITY

D-1 PROCEDURE

D-1.1 Take the material exposed to an atmosphere of 95 percent relative humidity and fill it in a 250 ml measuring cylinder up to 50 ml mark without knocking. Shake the sample down by gently lifting the cylinder as far as 5 cm from the base pad and releasing it on the table. Repeat this knocking for 4 more times. Add further 50 ml of the sample and knock five times, as above, after every 50 ml addition up to 250 ml. Level the cylinder with the material without any further knocking. Empty out the material from the cylinder and weigh to the nearest 0.5 g.

D-2 CALCULATION

Bulk density, g/l = $4M$

where

M = mass in g of the material in the cylinder.

ANNEX E

[*Table* 1, *item* (v) and *Clause* 7.1]

DETERMINATION OF INSOLUBLE MATTER

E-1 APPARATUS

E-1.1 Glass Three Piece Funnel, 7 cm in diameter.

E-1.2 Glass Filter Circled, grade GF/A, 7 cm in diameter.

E-1.3 Flask, 100 ml.

E-1.4 Weighing Bottles

E-1.5 Beakers

E-1.6 Vacuum Pump

E-2 PROCEDURE

E-2.1 Weigh 2 to 2.5 g of the sample and place into the flask, add 50 ml water, shaking flask whilst addition is taking place to ensure that the whole of the sample is thoroughly wetted out.

E-2.2 Allow to stand for about 1 hour, then gently warm to 55 to 60°C to dissolve the sample and make up to 100 ml.

E-2.3 The glass filter which has been previously dried in a weighing bottle at 105°C for 2 to 3 hours is weighed and placed in the three-piece funnel, vacuum applied and the sample solution is filtered. The flask and filter are washed thoroughly with 200 to 300 ml hot water. The filter is then removed and returned to the weighing bottle and allowed to dry at 105°C and then reweighed.

E-3 CALCULATION

$$\text{Insoluble matter, percent by mass} = \frac{m \times 100}{M}$$

where

m = increase in mass in g of glass filters, and

M = mass of sample in g taken for the test.

ANNEX F

[*Table* 1, *item* (vi) and *Clause* 7.1]

DETERMINATION OF TOTAL ASH

F-1 PROCEDURE

F-1.1 Weigh about 2.00 ± 0.01 g sample on dry mass basis in a tared platinum or procelain crucible. Add 3 ml distilled water, ignite on a low flame, cool and then add 1 ml of 1 : 1 sulphuric acid to the residue and dry again on burner. Keep in a muffle furnace at 800°C for 20 minutes (or at 600°C for about one hour). Weigh the crucible with residue after cooling in a desiccator. Repeat this procedure till a constant mass is obtained.

F-2 CALCULATION

F-2.1 Ash is assumed to be sodium sulphate (Na_2SO_4) and is then calculated as sodium oxide (Na_2O) as:

$$A = \frac{(d - b) \times 100 \times 0{\cdot}437}{C - b}$$

where

A = ash content, percent by mass;

d = mass in g of crucible plus ash;

b = mass in g of crucible; and

c = mass in g of crucible plus sample.

ANNEX G

[*Table* 1, *item* (vii) and *Clause* 7.1]

DETERMINATION OF DEGREE OF HYDROLYSIS

G-1 REAGENTS

G-1.1 Sodium Hydroxide Solution, 0.1 N and 0.2 N, prepared from analytical reagent grade sodium hydroxide and standardized by titration against sulphamic acid as given in **G-1.1.1.**

G-1.1.1 Correctly weigh 2 to 2.5 g of sulphamic acid which has been dried in a desiccator, and add water to make 250 ml. Use 25 ml of this solution and standardize the sodium hydroxide solution by using bromothymol as an indicator.

G-1.2 Sulphuric Acid Solution, 0.1 N and 0.2 N, prepared from analytical reagent grade sulphuric acid.

G-1.3 Phenolphthalein

G-1.4 Sulphamic Acid

G-2 APPARATUS

G-2.1 Erlenmeyer Flask with Ground Stopper, 300 ml capacity.

G-2.2 Burrette, 50 ml capacity, minimum scale value 0.1 ml.

G-2.3 Transfer Pippette, 25-ml capacity.

G-2.4 Chemical Balance, weighing capacity 100 to 200 g with an accuracy up to 1 mg or better.

G-3 PROCEDURE

G-3.1 Weigh accurately about 3 g of the sample in the Erlenmeyer flask with ground stopper, add about 100 ml of water and dissolve the sample by heating. After cooling to room temperature, add 25 ml of 0.1 N sodium hydroxide solution and keep the solution at room temperature for at least 2 hours. Then add 25 ml of 0.1 N sulphuric acid and titrate the excess acid with 0.1 N sodium hydroxide solution using phenolphthalein as indicator until a pale pink colour persists in the solution. Note the volume of 0.1 N sodium hydroxide solution used in the titration.

G-3.2 Cany out a blank titration separately as prescribed in **G-3.1** and note the volume of 0.1 N sodium hydroxide solution used in titration.

G-4 CALCULATIONS

G-4.1 Calculate the percentage mass of residual acetic acid radical (*A*) based on the pure component, the mole percent of residual acetic acid radical (*B*) and the degree of hydrolysis (*C*) correct to the two decimal places by the formulae:

$$A = \frac{0{\cdot}60 \times (a - b) \times F}{S \times P}$$

$$B = \frac{44{\cdot}05\,A}{60{\cdot}05 - 0{\cdot}42\,A}$$

and

$$C = 100 - B$$

where

a = volume of 0.1 N sodium hydroxide solution used in the titration with the sample (*see* **G-3.1**),

b = volume of 0.1 N sodium hydroxide solution used in blank titration (*see* **G-3.2**),

F = normality of 0.1 N sodium hydroxide solution,

S = mass of original sample in g, and

P = pure component percent of polyvinyl alcohol.

ANNEX H

(*Clause* 6.1)

SAMPLING OF POLYVINYL ALCOHOL FOR SIZING OF PAPER

H-1 GENERAL REQUIREMENTS OF SAMPLING

H-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

H-1.1 Samples shall not be taken at a place exposed to the adverse effects of weather.

H-1.2 Sampling instruments and sample containers shall be clean and dry.

H-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

H-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

H-1.5 The sample shall be placed in suitable, clean, dry and air-tight glass or other suitable containers on which the material has no action.

H-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

H-1.7 Each sample container shall be suitably stoppered and sealed air-tight after filling, and marked with full particulars of the material (*see* **5.2**), the date of sampling and the lot and batch number.

H-1.8 Samples shall be stored in a cool and dry place.

H-2 SCALE OF SAMPLING

H-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture and of the same grade shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each shall constitute separate lot.

H-2.1.1 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirements of the specification.

H-2.2 The number (*n*) of containers to be selected at random from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected from Lots of Different Sizes

Lot Size *N* (1)	Sample Size *n* (2)
Up to 15	3
16 „ 40	4
41 „ 110	5
111 „ 180	6
181 ,, 300	7
301 ,, 500	8
501 ,, 800	9
801 and above	10

H-2.3 These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS 4905 : 1968. In case this standard is not available, the following procedure shall be adopted :

> Arrange all the containers in the lot in a systematic manner and starting from any container count them as 1, 2, 3,. . . . . up to *r*, where *r* is the integral part *of N/n* (*N* and *n* being the lot size and the sample size, respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

H-3 TEST SAMPLES AND REFEREE SAMPLES

H-3.1 From each of the containers selected according to **H-2.3,** draw with an appropriate sampling instrument a representative portion of the material, sufficient for carrying out the tests specified under **H-4**. These shall constitute the individual samples.

H-3.2 From each of the individual samples, equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

H-3.3 The material in the individual and composite test samples shall be divided into three equal parts and transferred to separate bottles, scaled and labelled with full identification particulars of the sample (*see* **H-1.7**). The material in each such bottle shall constitute a test sample. Separate these test samples representing individual and the composite samples into three identical sets of test samples.

H-3.4 Distribution of Test Samples

One set of test samples shall be sent to the purchaser and another to the supplier. The third set of test samples bearing the seals of the purchaser and the supplier shall constitute the referee sample to be used in case of dispute between the purchaser and the supplier, and shall be kept at a place agreed to between the purchaser and the supplier.

H-4 NUMBER OF TESTS

H-4.1 Tests for determination of viscosity and degree of hydrolysis shall be carried out on individual samples.

H-4.2 Tests for determination of the remaining characteristics shall be performed on the composite sample.

H-5 CRITERIA FOR CONFORMITY

H-5.1 For Individual Samples

For those characteristics which are tested on individual samples, the means and range of test results shall be computed as follows:

$$\text{Mean } (\bar{X}) = \frac{\text{Sum of the individual test results}}{\text{Number of test results}}$$

Range ($\bar{R}$) = Difference between the maximum and minimum values of test results

H-5.1.1 The conformity criterion for viscosity, and degree of hydrolysis shall be that when $\bar{X} + 0.6\,R$ computed from the test results, it shall lie within the limits prescribed in Table 1.

H-5.2 For Composite Sample

For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample (*see* **H-4.2**), the test results shall comply with the corresponding specified values.

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